PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:	10/789,899
Filing Date:	February 27, 2004
Applicant:	Frederick E. Pinkerton et al.
Group Art Unit:	1754
Examiner:	Wayne A. Langel
Title:	MIXED HYDROGEN GENERATION MATERIAL
Attorney Docket:	GP-303644 (8540R-000058)

DECLARATION OF PRIOR INVENTION IN THE UNITED STATES TO OVERCOME CITED PATENT PUBLICATIONS PURSUANT TO 37 C.F.R. §1.131

PURPOSE OF DECLARATION

1. I am a co-inventor of the patent application identified above and of the subject matter described and claimed therein, including of Claims 1 through 52.

2. This declaration is being presented to establish conception and reduction to practice of the invention of the patent application identified above in the United States at a date prior to June 25, 2003.

FACTS & DOCUMENTARY EVIDENCE

3. Prior to June 25, 2003, having earlier conceived of the concept of storing hydrogen by reacting a nitride with a hydride, I submitted a record of invention to the legal department at General Motors Corporation. To establish the date of conception of the invention of the claims of this patent application, the attached record of invention document is submitted as Exhibit A. The redacted portions of Exhibit A either disclose dates that are all prior to June 25, 2003 or disclose personal confidential information. This document (hereinafter referred to as the "ROI"), was: prepared prior to June 25, 2003; identifies me as one of the co-inventors; and discusses and illustrates the conceived invention. The ROI includes

a description of an example of hydrogen storage compounds where hydrogen gas is released by reacting a nitride compound and a hydride compound to form one or more byproduct compounds. The ROI further includes summary pages and various graphs illustrating various embodiments of the invention described in the application, as well as hand-written lab note pages further evidencing conception of the invention.

4. Our invention was reduced to practice and experiments were conducted to generate data detailed in Figures 1 - 5 of the above identified patent application and in the lab notebook pages describing experimental details regarding nitride and hydride systems (attached as Exhibit B). The redacted portions of Exhibit B are dates that are all prior to June 25, 2003. The detailed description of the above identified patent application (at Paragraphs 35-46) details how the data was generated to create Figures 1-5, respectively. To establish the date of reduction to practice of the subject matter as claimed in this patent application, Exhibit B contains laboratory notebook information illustrating reduction to practice prior to June 25, 2003.

DECLARATION

5. As the person signing below I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE

Dated: Dec. 14, 2006

Martin S. Meyer

EXHIBIT A

37 C.F.R. §1.131 Declaration of Martin S. Meyer
U.S.S.N. 10/789,899 entitled "Mixed Hydrogen Generation Material."

Inter-Organization

GM

Research & Development and Planning

Date: [redacted]

Subj: R&D and Planning & NAPD Record of Invention File No. GP-303644

To: Kathryn A. Marra
GM Corporation Legal Staff
300 Renaissance Center
M.C. 482-C23-B21
P.O. Box 300
Detroit, MI 48265-3000

Attached is the Record of Invention entitled "Mixed Hydrogen Generation Material" in the name(s) of Frederick E. Pinkerton (430), Martin S. Meyer (430), and Gregory P. Meisner (430).

Carol E. Siino
Manager, Intellectual Property
M.C. 480-106-359
810/986-2520

Attachment

C: Materials & Processes (430)

REDACTED

CONFIDENTIAL AND PRIVILEGED

GENERAL MOTORS CORPORATION

File No. GP-303644

RECORD OF INVENTION

This Record of Invention must be completed with sufficient detail so that your invention can be understood and evaluated by both your engineering management and by a GM Legal Staff patent attorney. Novelty and competitive significance of your invention will be evaluated based on the information you provide.

Invention Title: Mixed Hydrogen Generation Material

Inventor #1

Name: Frederick (First Name) B. (Middle Initial) Pinkerton (Last Name) Citizen of: USA

Social Security No. [redacted] GM Employee: ☒ Yes ☐ No ☒ Salary ☐ Hourly ☐ Contract

Home Address: 52536 Bordeaux Way (Street) Shelby Township, Michigan (City and State) 48315 (Zip Code)

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Non-GM Employer: Phone No. (Area Code) + Number

Non-GM Employer Address: (Street) (City and State) (Zip Code)

*Inventor #2**

Name: Martin (First Name) S. (Middle Initial) Meyer (Last Name) Citizen of: USA

Social Security No. [redacted] GM Employee: ☒ Yes ☐ No ☒ Salary ☐ Hourly ☐ Contract

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Non-GM Employer Address: (Street) (City and State) (Zip Code)

* If there are more than two (2) inventors for this invention use the template at the end of this form.

[redacted]

File Number:

REDACTED

Answer questions 1 - 8, completing all of them to the best of your knowledge.

1. This invention was first thought of on: [redacted]

2. This invention has been or is expected to be disclosed outside GM on: _____

3. This invention has been used or is committed to be used in production on: _____

4. This invention has been offered for sale outside GM on: _____

5. Was this invention made while working on a Government Contract? ☐ Yes ☒ No

   If yes, identify the government Contract No. _____

6. Identify the product or process in which the invention is incorporated: Hydrogen Storage, Fuel Cells

7. List all individuals who can provide information about the making of the invention. This list may include individuals who made the first sketch, description, or tests and individuals who are familiar with the facts relating to the making of the invention.

   Frederick E. Pinkerton, Martin S. Meyer, Jan F. Herbst, John Vajo, Gregory P. Meisner, Michael P. Balogh

8. Each inventor has a legal duty to disclose all information known that is material to patentability of this invention. Such information includes the relevant prior art, which may be in the form of current or past products, equipment, processes, materials, patents, publications, advertisements, displays, and unpublished developments and proposals—whether originated by you, others in GM, competitors, suppliers, customers or others. Such information also includes disclosure of this invention outside GM, sales and offers of products using this invention, use of this invention in production and disputes about who should be considered as an inventor of this invention. To comply with the duty to disclose, list here and attach a copy of all such information, to the extent known.

   LiBH4 is known to be a "hydrolysis hydride" which will release hydrogen on exposure to water. Considerable work has been done on this material by Scott Jorgensen and collaborators at GM R&D and by other investigators elsewhere.
   A previous record of invention, GP-302578, has been submitted on reversible hydrogen storage in the Li-N-H system according to the formula $LiNH_2 + LiH <-> Li_2NH + H_2$.
   A Microsoft PowerPoint presentation authored by John Vajo described his work at HRL on several coupled hydride compounds, namely LiOH+LiH, LiOH+NaH, LiH+Si, and MgH_2+Si. He has also previously discussed $LiBH_4$+MgH_2.
   Greg Meisner has previously examined the mixtures $LiAlH_4 + LiNH_2$ and $LiAlH_4 + 4\ LiNH_2$ for the purpose of using the LiH produced from decomposition of $LiAlH_4$ to combine with the $LiNH_2$ as above.

REDACTED

Answer question 9 thoroughly.

9. Describe the invention in sufficient detail so that its nature, operation and usefulness can be understood. (Attach drawings, diagrams and further description, when necessary. Additional guidelines are listed below.)

See attached.

Mechanical and Electrical Devices: Include illustrations assigning reference numbers to the main elements and refer to the reference numbers in a description that explains how the main elements are connected or related and how they operate.

Electrical Circuits and Controls: Include circuit diagrams and a functional description.
Computer Software and Manufacturing or Business Processes: Include a flowchart or other step-by step overview.

Chemical Inventions: Identify all essential materials used, and alternatives therefor, in chemical terms – not tradenames. Identify and quantify all significant variables (e.g. temperature, pressure, concentration, pH etc.) of the process or material specifying operating ranges and the preferred example. Discuss the significance of each variable. Provide a recipe for at least one working example of the invention.

[redacted] File Number: _____ REDACTED

I hereby assign this invention to General Motors Corporation and authorize General Motors Corporation to file an application on my behalf.

Frederick E. Pinkerton — Frederick E. Pinkerton — [redacted]
INVENTOR – SIGNATURE (ALSO, PRINT NAME) DATE

Martin S. Meyer — Martin S. Meyer — [redacted]
INVENTOR – SIGNATURE (ALSO, PRINT NAME) DATE

Gregory P. Maine — GREGORY P. MELSNER — [redacted]
INVENTOR – SIGNATURE (ALSO, PRINT NAME) DATE

This invention was reviewed and understood by me:

Jan F. Herbst — Jan F. Herbst — [redacted]
1st WITNESS – SIGNATURE (ALSO PRINT NAME) DATE

Michael P. Balogh — Michael P. Balogh — [redacted]
2nd WITNESS – SIGNATURE (ALSO, PRINT NAME) DATE

REDACTED

[redacted] File Number: _____

Answer the following questions if helpful in describing this Invention

10. What benefits will be realized by using this invention?

The benefit of this invention is as a source of hydrogen for fuel cell applications.

11. What is the state of development of this invention?

Hydrogen generation has been successfully demonstrated in $LiBH_4 + 2\ LiNH_2$ with an onset temperature of 100 °C and bulk hydrogen release at a temperature of 245 °C.

12. To the extent known, what alternatives exist for accomplishing substantially the same result as this invention?

Hydrogen can be stored as a compressed gas, as a cryogenically cooled liquid, or in a solid. Current solid storage media lack hydrogen capacity, have slow kinetics, or require high or low temperatures. Hydrogen release in $LiBH_4$ can be accomplished by addition of water or water vapor, but with a weight penalty due to the additional weight of the water.

13. Describe the background of the invention. This description may include the state of the prior art and may identify deficiencies in the prior art that are overcome by this invention.

A survey of the literature revealed the existence of Li_3BN_2; this invention resulted from considering Li_3BN_2 as the reaction product of a mixture of other hydrogen containing compounds, such as $LiBH_4 + LiNH_2$, thereby releasing hydrogen gas.

The binary couple $LiNH_2 + LiH$ has been the subject of a previous Record of Invention.

Conventionally, $LiBH_4$ is known as a hydrolysis hydride, in which hydrogen is released by exposure to water.

Thermal decomposition of $LiBH_4$ is impractical, requiring a temperature of ~400 °C, and at that temperature it releases B-H compounds in addition to H_2.

REDACTED

[redacted]

File Number: _____

Inventor # 3

Name: Gregory (First Name) P. (Middle Initial) Meisner (Last Name) Citizen of: USA

Social Security No. _____ GM Employee: ☒ Yes ☐ No ☒ Salary ☐ Hourly ☐ Contract

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Non-GM Employer: _____ Phone No. _____ (Area Code) + Number

Non-GM Employer Address: _____ (Street) _____ (City and State) _____ (Zip Code)

Inventor #

Name: _____ (First Name) _____ (Middle Initial) _____ (Last Name) Citizen of: _____

Social Security No. _____ GM Employee: ☐ Yes ☐ No ☐ Salary ☐ Hourly ☐ Contract

Home Address: _____ (Street) _____ (City and State) _____ (Zip Code)

GM Unit: _____ GM Phone No. (8)- _____ (Centrex Number) _____ (Area Code) + Number

GM Address: _____ Mail Code: _____ FAX Number: (8)- _____ (Centrex Number)

Non-GM Employer: _____ Phone No. _____ (Area Code) + Number

Non-GM Employer Address: _____ (Street) _____ (City and State) _____ (Zip Code)

REDACTED

[redacted] File Number: _____

This invention describes an example of coupled hydrogen storage compounds in which hydrogen gas is released by heating a mixture of two compounds, at least one of which contains hydrogen. The chemical reaction considered here is given by:

$$LiBH_4 + 2\,LiNH_2 \rightarrow Li_3BN_2 + 4\,H_2$$

The theoretical amount of hydrogen released is 11.8 wt% of the starting hydride mixture.

Hydrogen release has been demonstrated as shown in the accompanying figures.

In the first experiment, a mixture of $LiBH_4$ and $LiNH_2$ of molar ratio 1:2 was used according to the above chemical reaction formula. The mixture was ground together in an inert atmosphere using an agate mortar and pestle. A sample weighing 1.102 grams was loaded into the sample holder of the PCI apparatus and all the spaces were evacuated. Then the temperature was increased and pressure measurements were recorded until the sample temperature reached ~400°C. Figure 1 shows the sample temperature and hydrogen desorption versus time. We note a slight decrease in the gas pressure between 10 and 25 hours, Figure 2, which could indicate a small amount of re-absorption of the desorbed gas. The pressure of the desorbed gas reached about 900 kPa in this experiment corresponding to a hydrogen weight per cent desorption of about 3.06 wt%. Subsequently, the sample was cooled to ~50°C and the desorbed gas evacuated. A second heating cycle was performed, Figure 3, and another small amount of gas desorbed. Figure 4 shows a composite of the two experimental results showing a total desorption of 3.21 wt% hydrogen. The onset of the desorption as a function of temperature, shown in Figure 5, is approximately 100°C. Finally, we attempted to absorb hydrogen back into this sample by pressurizing up to >9000 kPa at 200°C. Figure 6, however, shows no hydrogen uptake to within the experimental uncertainty of the PCI apparatus. Subsequent x-ray diffraction analysis, Figure 7, revealed that this sample was heavily oxidized and contained mostly Li_2O and Li_3BO_3.

In the second experiment, $LiBH_4$ and $LiNH_2$ were mixed in a 1.18:2 molar ratio (the excess $LiBH_4$ is the result of a weighing error) and ball milled for 10 minutes in a steel vial using a SPEX 8000 mixer/mill. Some of the resulting powder was placed into a thermogravimetric analyzer (TGA), where hydrogen release appears as weight loss of the sample as shown in the upper panel of Figure 8. The mixture was heated in stages up to 245°C under 1.3 atm flowing He gas, and at temperatures > 200°C lost weight totaling 13.5 wt%. A mass spectrometer operated as a residual gas analyzer (RGA) was used to monitor the composition of the exhaust gas, as shown in the lower panel of Figure 8. The broad humps in the RGA signal from all species during heating reflect a general change in the background signals of all the species related to heating the TGA, and these are unrelated to the sample. Alone among them, H_2 gas shows a large excess concentration strongly correlated with heating events. The sharp drop in the H_2 mass spectrometer signal at the end of the TGA weight loss is particularly dramatic. A semi-quantitative analysis of the amount of H_2 released gives 11.8 wt% (the exact correspondance with the chemistry above must be regarded as somewhat fortuitous). X-ray diffraction analysis of the starting ball-milled mixture is shown in Figure 9. The red bars indicate the expected

diffraction lines from $LiNH_2$ and the green bars are the expected lines from $LiBH_4$. The presence of large diffraction lines, particularly at 2θ ~16°, ~21°, and ~29°, shows that ball-milling has largely transformed the starting compounds into another compound that we have not yet been able to identify. A minor amount of Li_2O is also present as an impurity, and it is most likely an inert diluent. Figure 10 shows the x-ray diffraction pattern obtained after desorption of >10 wt% hydrogen in the TGA. The dominant phase is the expected Li_3BN_2 compound, but one or more additional phases (along with the impurity Li_2O phase) are also present, as indicated by the as yet unidentified extra diffraction lines. In contrast to the first sample, this sample after desorption is not heavily oxidized.

By itself $LiBH_4$ melts at ~280°C, but does not decompose with significant weight loss until ~400°C. By itself $LiNH_2$ decomposes slowly at 200°C and above, and at rates comparable to those observed in the example, but it does so by releasing ammonia rather than H_2. If this mechanism were responsible for the weight loss, the total loss should be 25 wt%. The example shows that when the two materials are combined via ball milling, the mixture decomposes by H_2 release at ~245°C.

Preliminary attempts to reverse the reaction, thereby providing a reversible hydrogen storage medium, were not successful. Efforts to make the reaction reversible continue, however. Incorporating a catalyst is one method known to both reduce the hydrogen release temperature and facilitate reabsorption in other hydrogen storage materials, and we expect that it will work for this invention as well.

A number of similar reactions can be written

$LiAlH_4 + 2\ LiNH_2 \rightarrow Li_3AlN_2 + 4\ H_2$ (9.5 wt%)
$NaBH_4 + 2\ NaNH_2 \rightarrow Na_3BN_2 + 4\ H_2$ (6.9 wt%)
$2\ LiBH_4 + Mg \rightarrow MgB_2 + 2\ LiH + 3\ H_2$ (8.9 wt%)
$Mg(BH_4)_2 + 3\ Mg(NH_2)_2 + 2Mg \rightarrow 2\ Mg_3BN_3 + 10\ H_2$ (7.4 wt%)
$Mg(BH_4)_2 + 3\ Mg(NH_2)_2 + 2MgH_2 \rightarrow 2\ Mg_3BN_3 + 12\ H_2$ (8.7 wt%)
$Mg(BH_4)_2 + 6\ MgNH \rightarrow 2\ Mg_3BN_3 + Mg + 7\ H_2$ (4.8 wt%)
$Mg(BH_4)_2 + 6\ MgNH \rightarrow 2\ Mg_3BN_3 + MgH_2 + 6\ H_2$ (4.1 wt%)
$Mg(BH_4)_2 + 2\ Mg(NH_2)_2 \rightarrow Mg_3B_2N_4 + 8\ H_2$ (9.6 wt%)

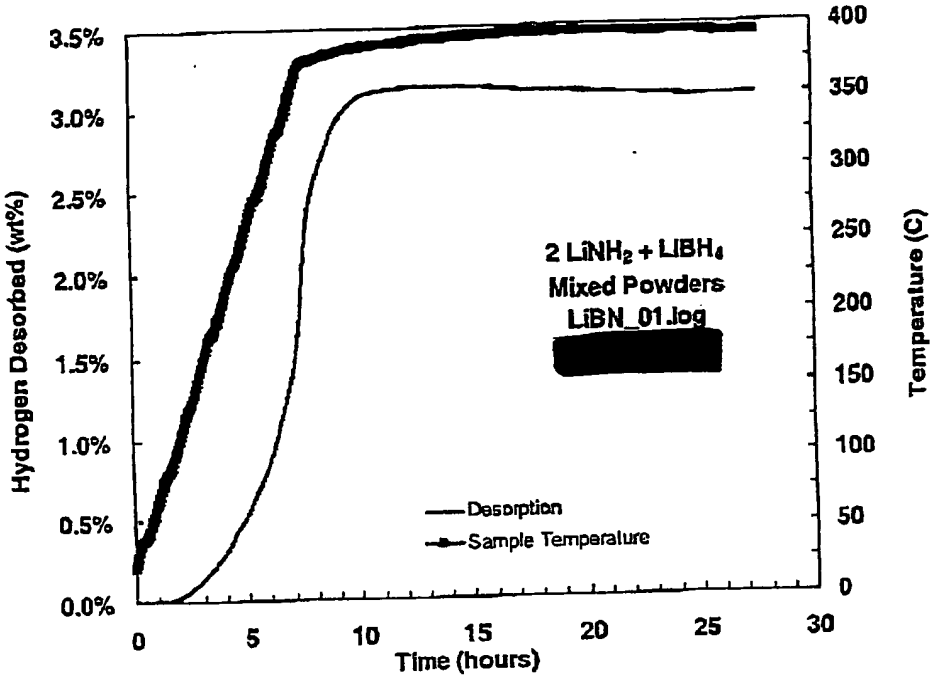

Figure 1

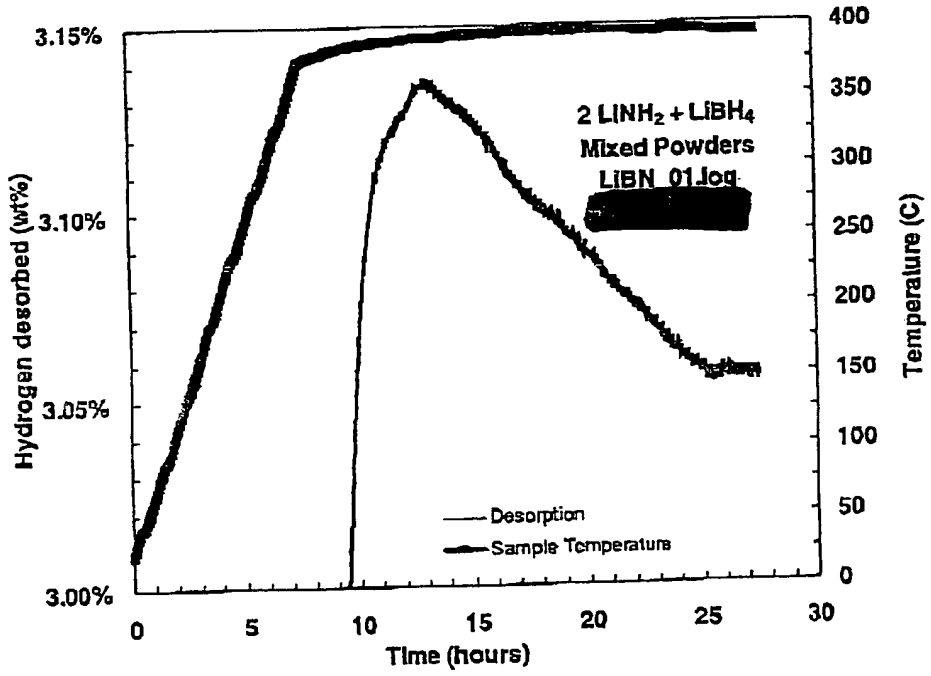

Figure 2

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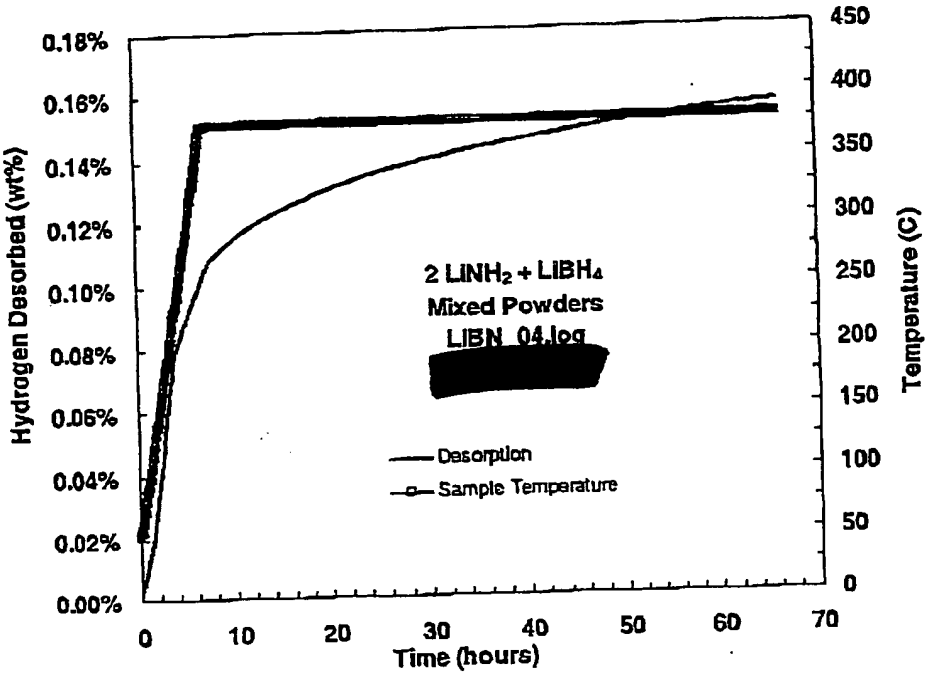

Figure 3

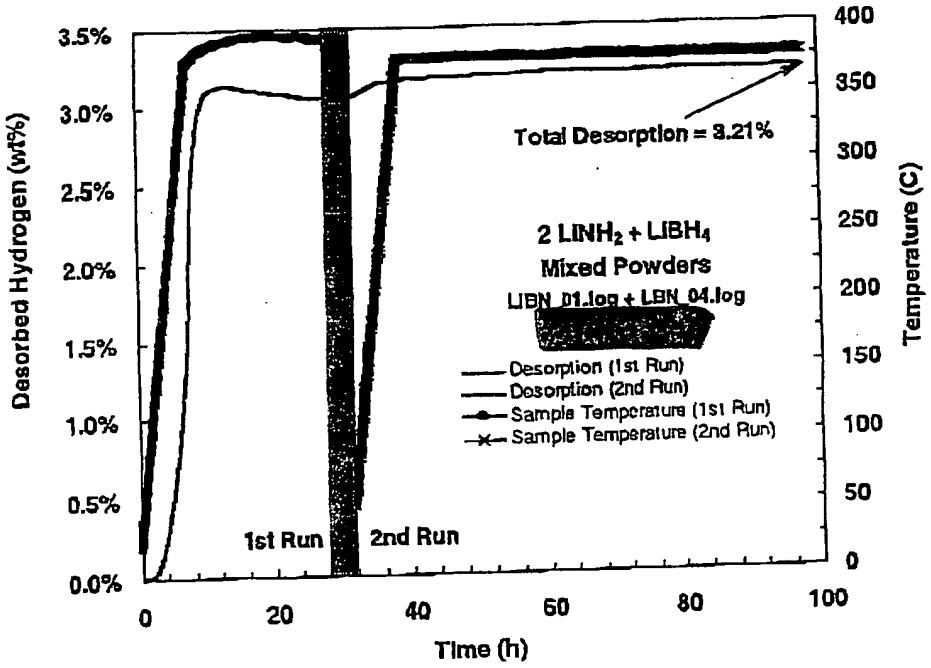

Figure 4

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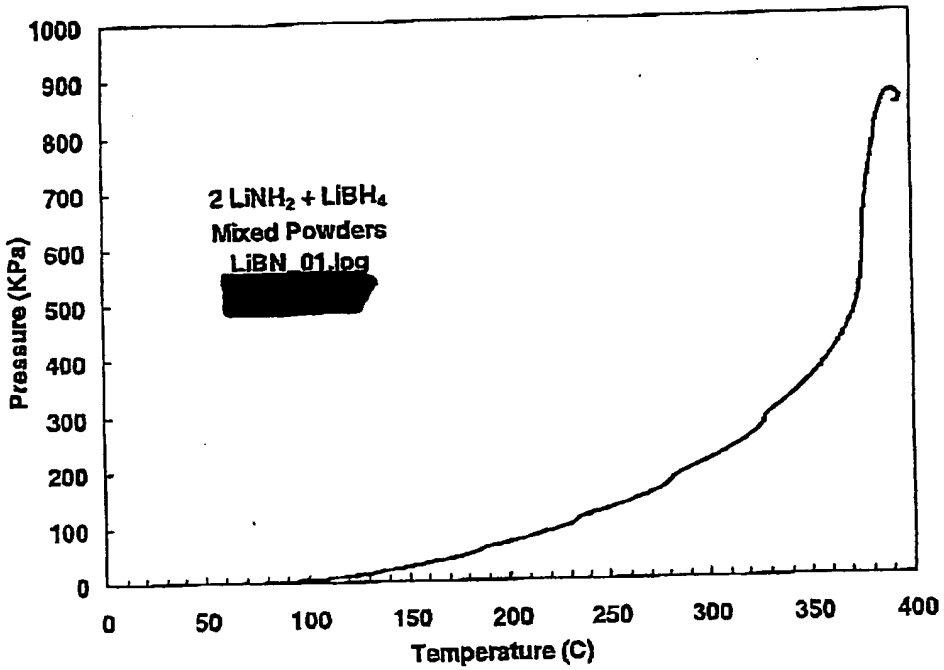

Figure 5

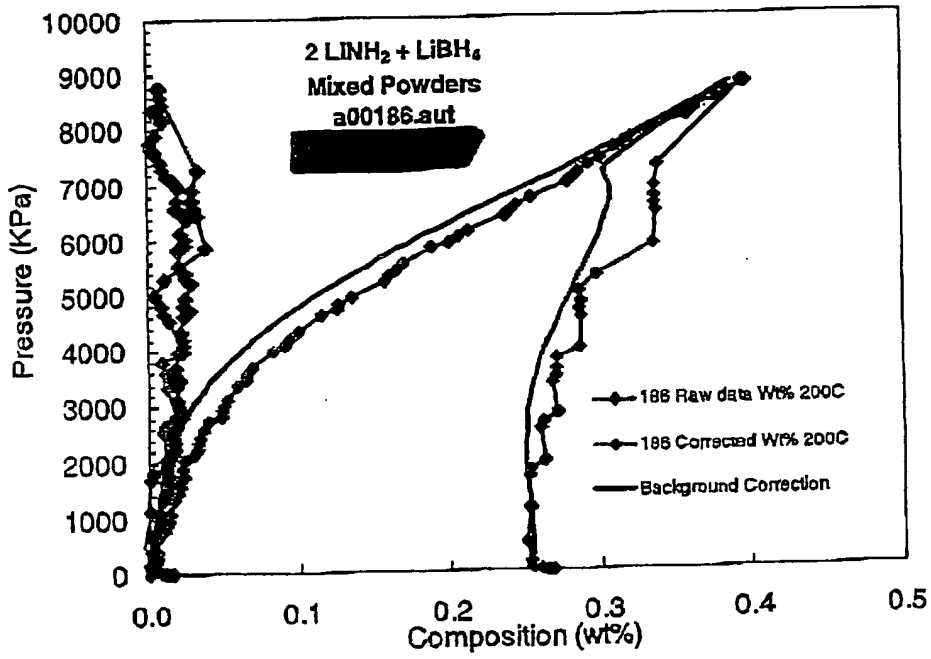

Figure 6

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er[redacted][001]

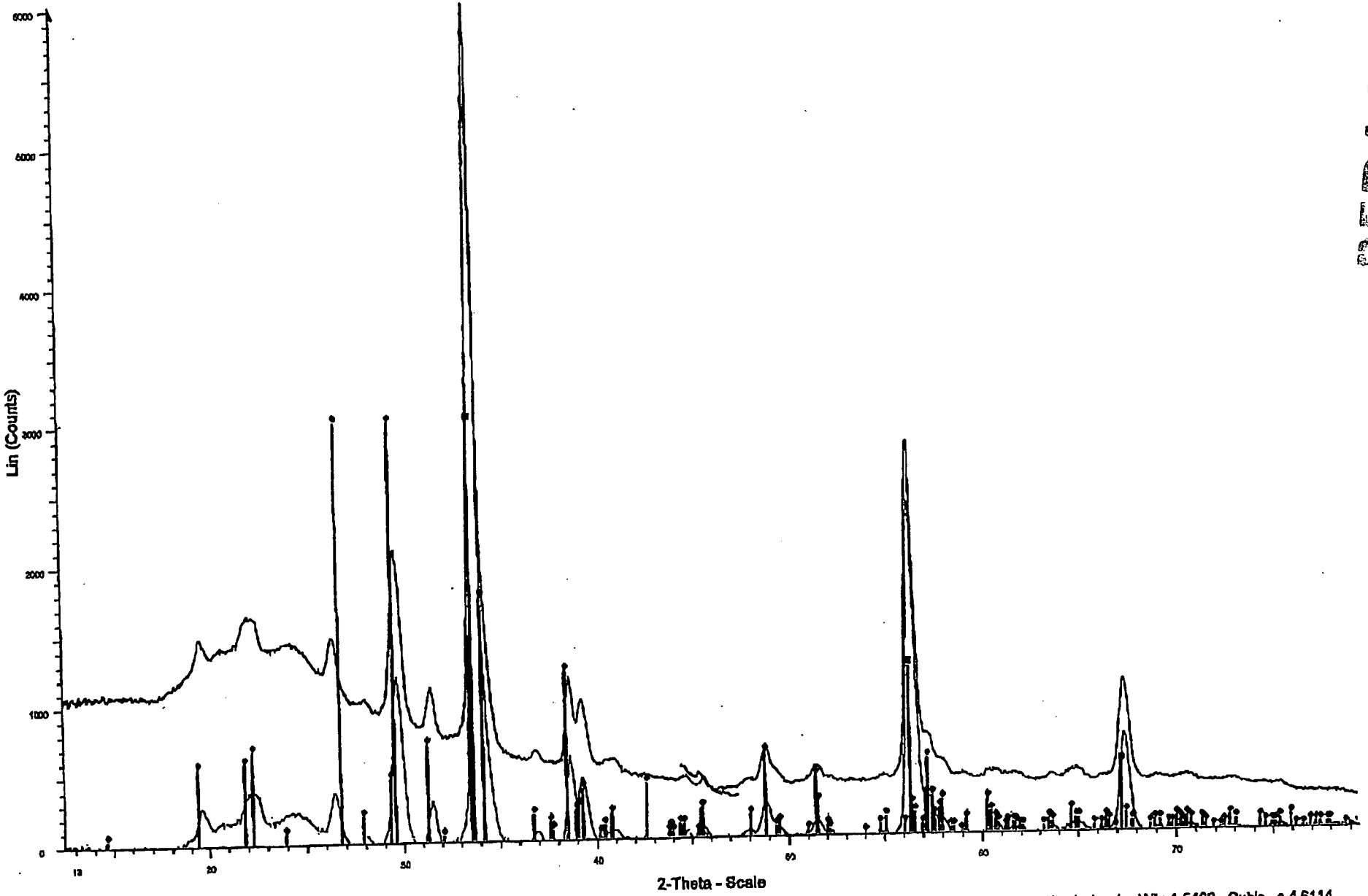

FIGURE 7

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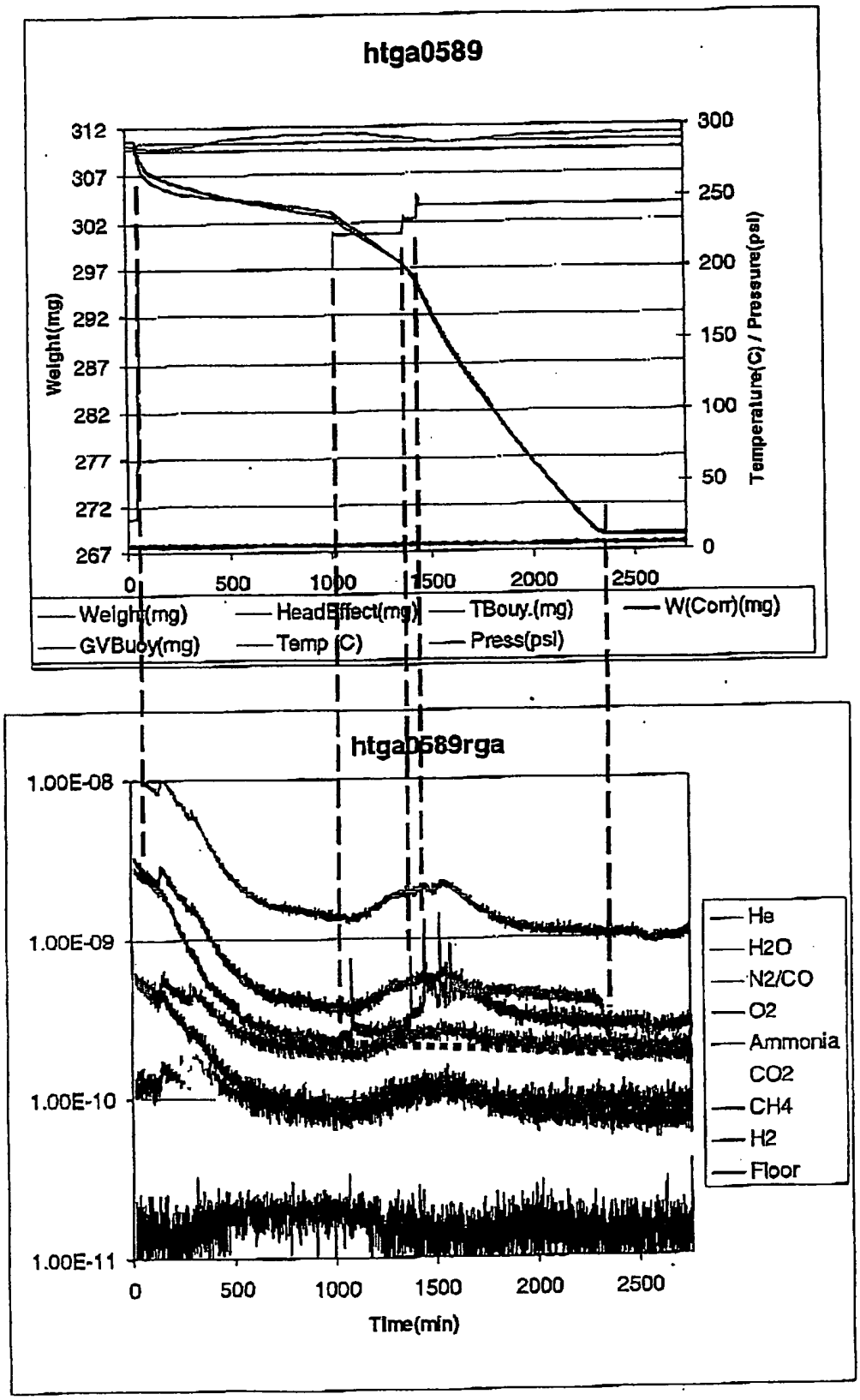

Figure 8

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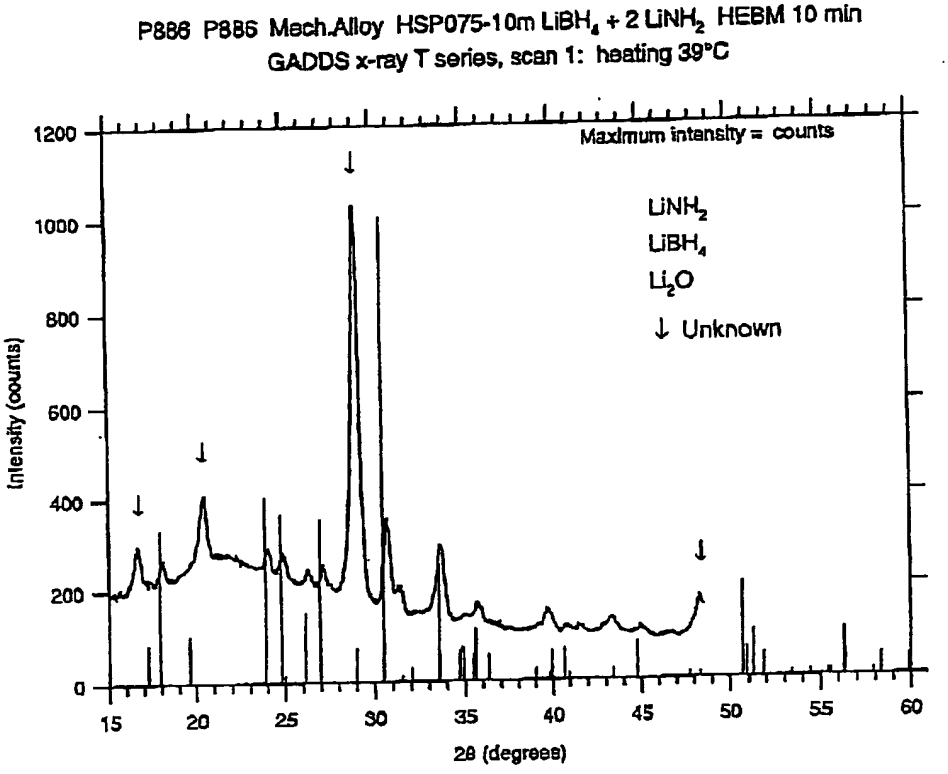


Figure 9

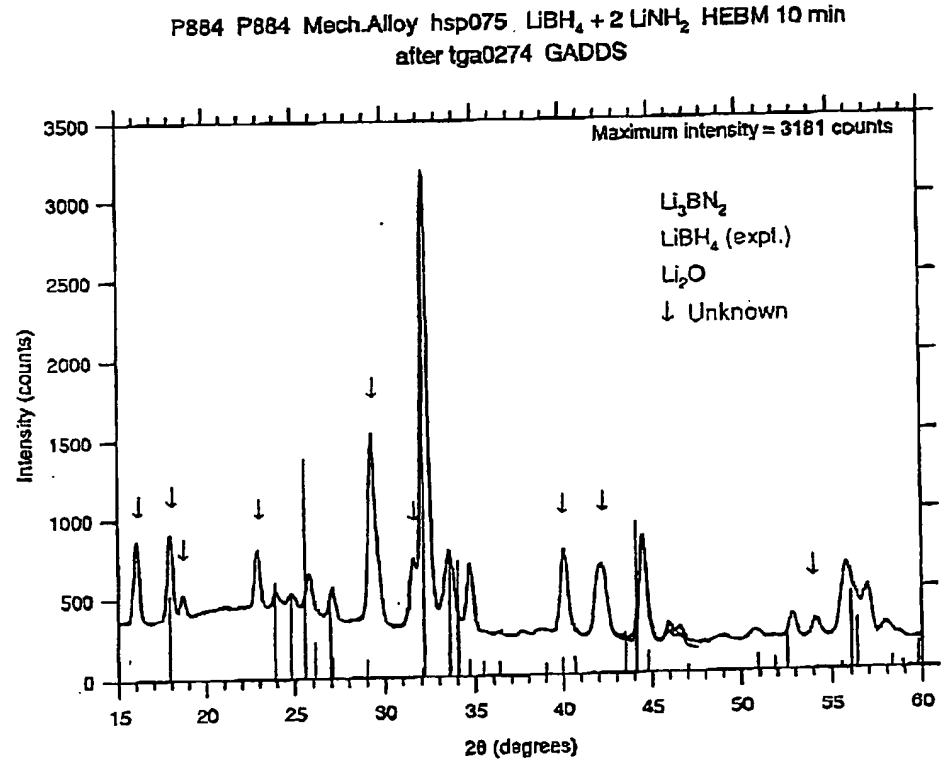


Figure 10

EXHIBIT B

37 C.F.R. §1.131 Declaration of Martin S. Meyer
U.S.S.N. 10/789,899 entitled "Mixed Hydrogen Generation Material."

DATE [redacted]

~~For PCT: 300 sec 8500 kPa max — turn set point up 1°C~~
~~High resolution LiAlO$_2$ $T_H = 265$~~
~~$T_S = 202.4°C$~~
~~a00184 art~~

[redacted]

~~Unload Sample: final wt: 10.466 Sample + Bucket~~
~~9.954 Bucket~~
~~0.512 gone~~

~~0.632 before~~
~~0.512 after~~
~~0.120~~

~~$\frac{0.120}{0.632} = 19\%$ loss~~

~~But lost some powder due to evacuation (on holder)~~

[redacted]

Idea for Li-B-N-H storage material

~~phase exists~~ Li_3BN_2 ~~(tP12) $P4_22_12$ #94~~

$a = 0.46435$ nm $= b$ $c = 0.52592$ nm

~~LT phase (below 1135K)~~
273
862°C

B	2a	0	0	0
Li1	2b	0	0	1/2
Li2	4d	0	1/2	0.25
N	4f	0.2962	0.2692	1/2

a). ~~Mix $Li_3N + BN = Li_3BN_2$~~

b) $2LiNH_2 + LiBH_4 = Li_3BN_2 + 4H_2$

does this reaction happen?

REDACTED

WITNESSED

WITNESSED

SIGNATURE Greg Meisner [redacted]

DATE [redacted]

$LiNH_2$: 6.941 + 14.0067 + 2(1.0079) = 22.9635 g/mole

$LiBH_4$: 6.941 + 10.81 + 4(1.0079) = 21.7826 g/mole

$\frac{4(1.0079)}{2(22.9635)+21.7826} \times 100 = 11.9085$ wt%

$\frac{2 \text{ moles } LiNH_2 \times 22.9635 \text{ g/mole}}{1 \text{ mole } LiBH_4 \times 21.7826 \text{ g/mole}} = 2.10843 \frac{g\ LiNH_2}{g\ LiBH_4}$

$LiBH_4$ 0.792 gms in Al_2O_3 crucible, out gas @ 125°C

final wt: (@ 180°C) (0.737 g)

.734 + .018 tare = 0.744 gms $LiBH_4$

⇒ 1.568 gms $LiNH_2$ req'd grind together in agate MEP

store in glass vial w/ screw lid

[redacted]

9.954 Sorbent wt: tare: 11.055 gms

1.102 Sample wt

11.056 total load into holder / on PCT

evacuate slowly, logfile LiBN_01 log 30 sec

in vac valves 5, 6, 7 open, all others closed

increase set point ~1°C/min to 50°C

REDACTED

WITNESSED _____

WITNESSED _____ SIGNATURE [signature]

Frederick E. Pinkerton Log #11

DATE [redacted]

Summary of new chemistries:

Listed in the email are:

(1) ⊛ $LiBH_4 + 2\,LiNH_2 \rightarrow Li_3BN_2 + 4\,H_2$ (11.8 wt%)

(2) ⊛ $2\,LiBH_4 + Mg \rightarrow MgB_2 + 2\,LiH + 3\,H_2$ (8.85 wt%)

The second of these, involving Mg, was subsequently discussed in a meeting between John Vajo and [redacted], along with a Florian Mertens on [redacted], along with a variety of other couples; including $2\,LiBH_4 + MgH_2$

In addition:

Li_3AlN_2 is also listed in the ternary database, so the Al equivalent is possible:

(3) ⊛ $LiAlH_4 + 2\,LiNH_2 \rightarrow Li_3AlN_2 + 4H_2$ (9.54 wt%)

The ternary phase diagram database does not contain entries for Na-B-N, Na-Al-N, Mg-B-N or Mg-Al-N However, the JCPDS x-ray database has a calculated pattern for $Na_3(BN_2)$ monoclinic. If this phase exists then a possible reaction is:

(4) $NaBH_4 + 2\,NaNH_2 \rightarrow Na_3BN_2 + 4\,H_2$ (6.9 wt%)

The phase $NaBN_{13}$ is also listed, but rated Q (questionable).

The JCPDS database lists Mg_3BN_3 as a known phase. Equations with ~~either~~ $Mg(BH_4)_2$ ~~or with~~ combined with either Mg amide $Mg(NH_2)_2$ or Mg imide MgNH proved impossible to balance unless excess Mg was introduced:

WITNESSED J. F. Herbst [redacted]

WITNESSED Mata L. Mo [redacted] SIGNATURE Frederick E. Pinkerton

REDACTED

DATE [redacted]

(7.37 wt%)

⑤ $2Mg + Mg(BH_4)_2 + 3Mg(NH_2)_2 = 2Mg_3BN_3 + 10H_2$

⑥ $2MgH_2 + Mg(BH_4)_2 + 3Mg(NH_2)_2 = 2Mg_3BN_3 + 12H_2$ (8.71 wt%)

From Mg imide:

(4.83 wt%)

⑦ $Mg(BH_4)_2 + 6MgNH = 2Mg_3BN_3 + Mg + 7H_2$

⑧ $Mg(BH_4)_2 + 6MgNH = 2Mg_3BN_3 + MgH_2 + 6H_2$ (4.14 wt%)

Reactions 5 + 6 are 3-component reactions, and thus may be difficult or slow in practice.

JCPDS also lists $Mg_3B_2N_4$, but as Q (questionable). If this phase exists we could have

⑨ $Mg(BH_4)_2 + 2Mg(NH_2)_2 = Mg_3B_2N_4 + 8H_2$ (9.61 wt%)

Pearson's does not list any phases not already discussed. There are no listings for Nd-Al-N or Mg-Al-N phases in the databases. If such unknown phases were to exist, Al cognates might be possible.

Note on reaction ③: Greg Meisner tried a related reaction

$LiAlH_4 + LiNH_2 \rightarrow \frac{1}{3}Li_3AlH_6 + \frac{2}{3}Al + H_2 + LiNH_2$

$\rightarrow LiH + Al + \frac{3}{2}H_2 + LiNH_2$

$\rightarrow Li_2NH + Al + \frac{5}{2}H_2$ (i.e. used LiH from $LiAlH_4$ to produce more H_2)

He did not report any unusual behavior.

REDACTED

WITNESSED J. F. Herbst [redacted]

WITNESSED [redacted] SIGNATURE [redacted]

Martin S. Meyer Log - 1 61

DATE [redacted]

Other mix F. Dubucha $LiBH_4 + 2LiNH_2 \rightarrow Li_3N_2B + 4H_2$ (11.8 wt%)

Sample hsp075 ball mill 10 minutes

$(6.94 + 10.81 + 4) + 2(6.94 + 14.0 + 2)$

21.75 + 2(22.94)

Mix up H_2 gen — 21.7 g + 45.88 g = 67.59

32 mg 190.5 $LiBH_4$ Lancaster Lot 10019317 — 32 wt% + 67.9 wt%

69 mg 375 $LiNH_2$ HSP074 2hr Ball milled

→ Ball mill 10 min in Steel vial 5

w/ flow
46.888
~~47.032~~ mg

Ztga 0274 .tsd — Zero TGA empty pan

Flowing Ar 20 Ar 25 to balance — Weight with sample 6.693 mg 30°C

Dirty melt point of $LiNH_2$ ~380°

Or 168%? $LiBH_4$ ~275 d

Note Furnace/Pan look off center

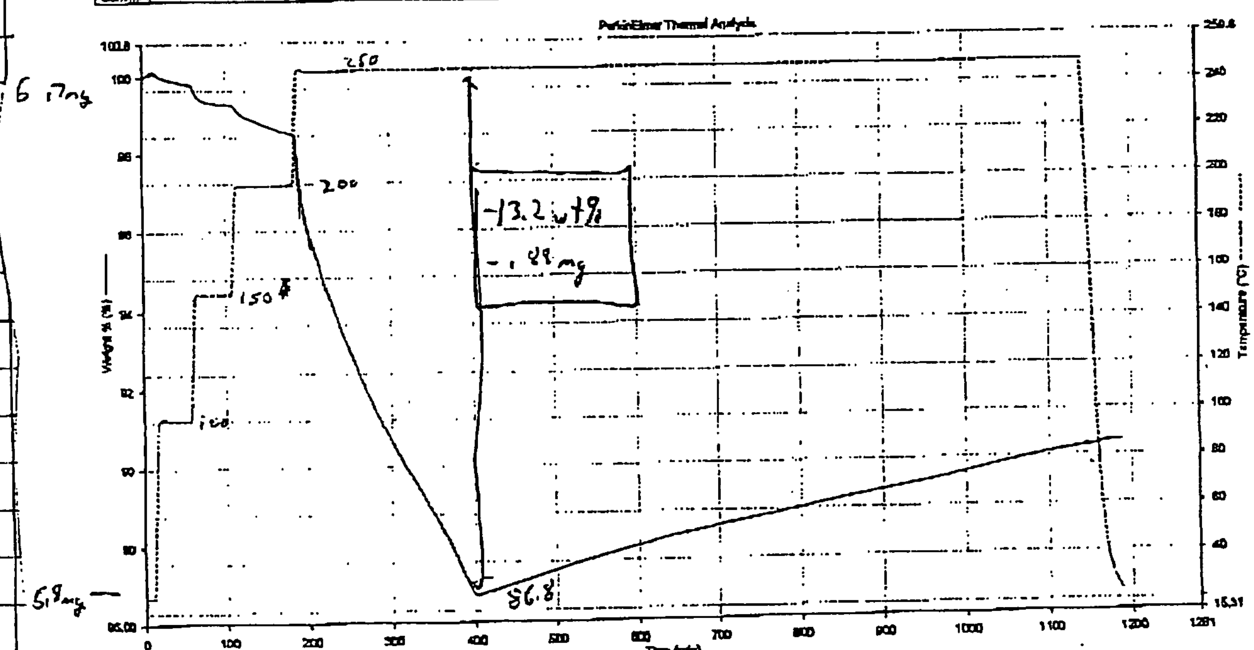
@ 250 H_2? carrying off during load bearing

Slope change @ 400 minutes? gas flow OK

X-RAY GADDS P 884 after Ztga 027

250° ambient Ar

Filename: C:\PEData\TGA\tga0274.tsd
Operator ID: MSM
Sample ID: hsp075 LiBH4 + 2(LiNH2)
Sample Weight: 6.693 mg
Comment:



[redacted]

1) Hold for 10.0 min at 30.00°C
2) Heat from 30.00°C to 100.00°C at 10.00°C/min
3) Hold for 40.0 min at 100.00°C
4) Heat from 100.00°C to 150.00°C at 10.00°C/min
5) Hold for 45.0 min at 150.00°C
6) Heat from 150.00°C to 200.00°C at 10.00°C/min
7) Hold for 70.0 min at 200.00°C
8) Heat from 200.00°C to 250.00°C at 10.00°C/min
9) Hold for 905.0 min at 250.00°C
10) Cool from 250.00°C to 0.00°C at 10.00°C/min
11) Hold for 20.0 min at 0.00°C

REDACTED

WITNESSED

SIGNATURE Martin S. Meyer [redacted]

WITNESSED

DATE htga0588.ctd

[redacted] Sample Tare

18t.256 = 1.534cc 1.449cc

310.2mg .8 gtgs

2 3/8 psi He

hsp025 LiBH4 + 2(LiNH2)

density 0.66 g/cc (Li 48%, Li 2B13 wt%)

= 1.21 g/cc

0) P(He) 3.3%, R(He) 3.3%, F(He) 3.3% 5psi ; no change

34) P (He) 15%, R (He) 3.3%, F(He) 3.3%

42) P (He) 3.3%, R (He) 3.3%, F(He) 3.3%

48) P (He) 3.3%, R (He) 15%, F(He) 3.3%

54) P (He) 3.3%, R (He) 3.3%, F(He) 3.3%

6..

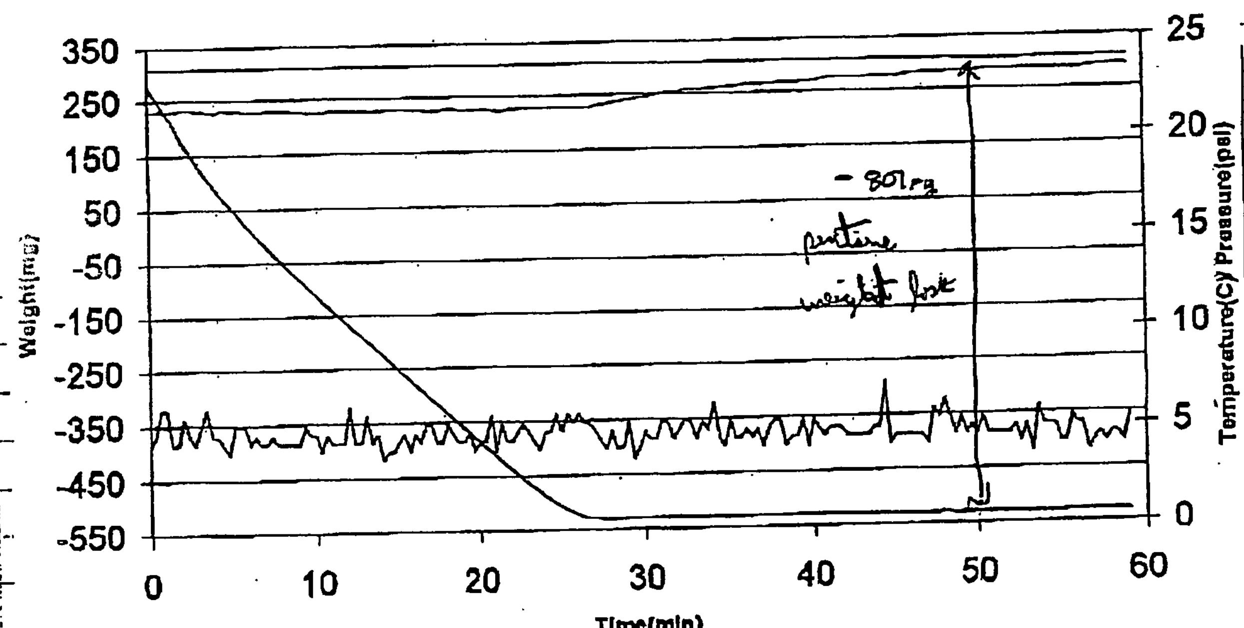
Tare Vol(cc)	Starting Gas GMW	Samp Vol(cc)	Starting Gas GMW
1.449	4	1.534	4
	1.262	1.271	

Velocity correction is for this Bucket diameter only!
Bucket Dia.(mm)
19

Offset(roughly starting wt. mg)
310.2
htga Mass (grams)
0.3
Head Effect Params.
m(@RTP) b
-0.19 0
-0.169724 at RTP

Theoretical m (htga M)->

htga0588



Weight(mg) HeadEffect(mg) TBouy.(mg) W(Corr)(mg)
GVBuoy(mg) Temp (C) Press(psi)

REDACTED

WITNESSED ____________

WITNESSED ____________ SIGNATURE ____________

DATE htga0589. ctd .25c [illegible] hsp075 4BH4 + 2 LiNH2 contd

[redacted]

Sample Tare

1.534cc 1.449cc

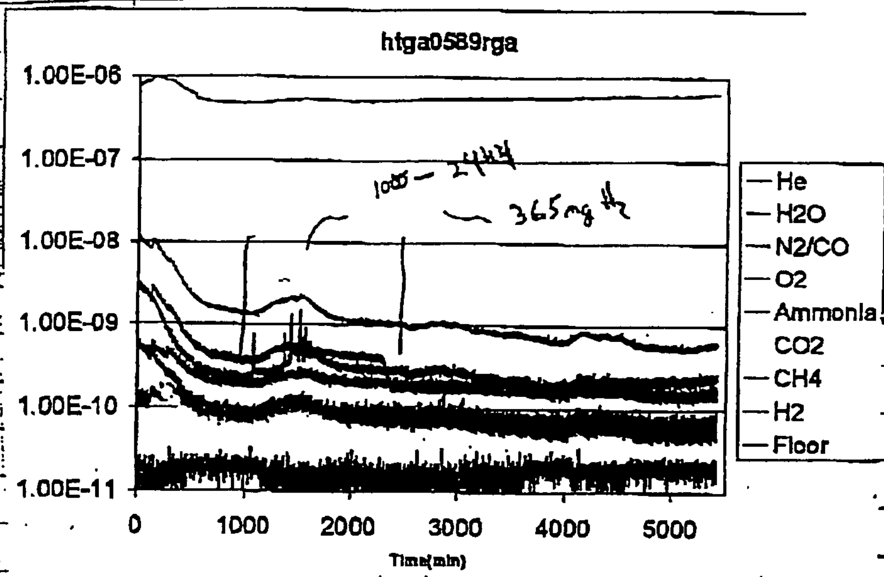
310.2mg -.06149 g htga

2.50 5 He

0) P(He) 3.38, R(He) 3.38, F(He) 3.38 5 psi

13) START RGA [illegible]

5.8% 269.4 mg -.10229 g htga



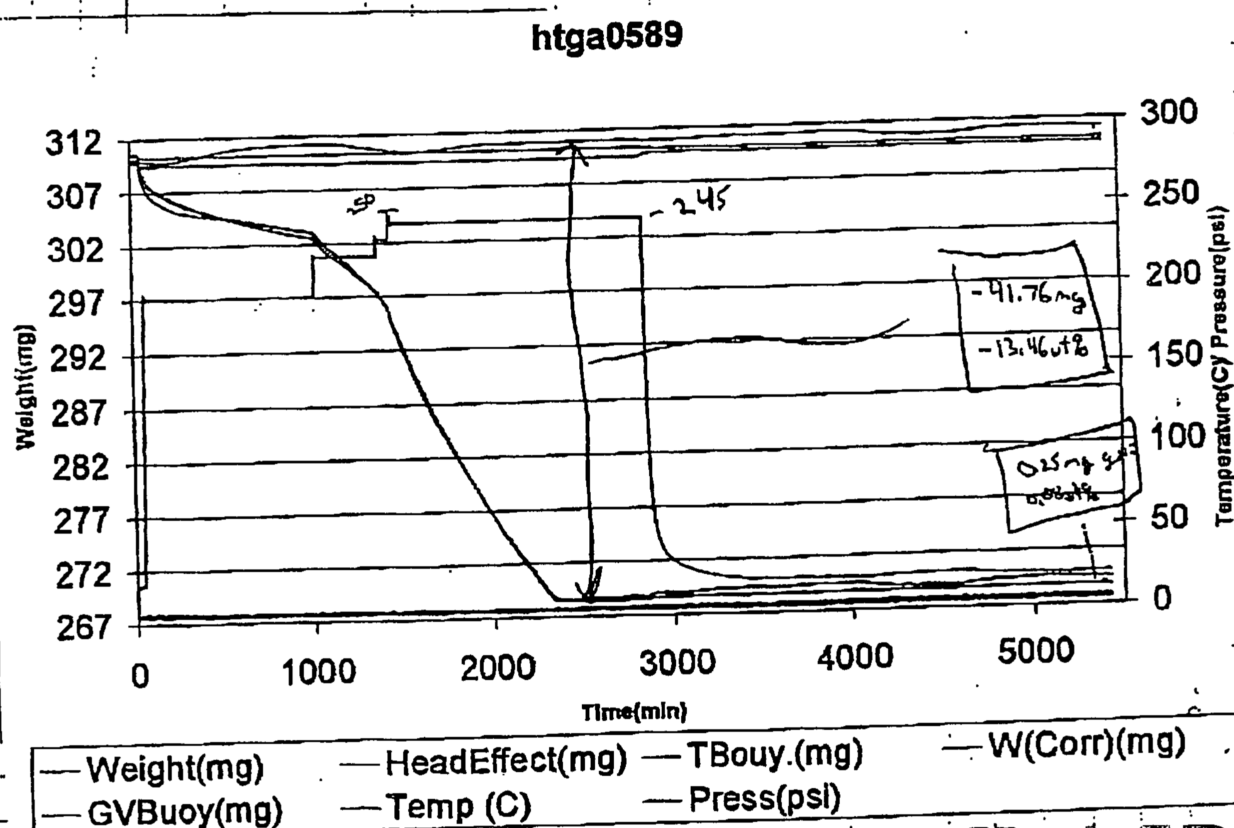
Tare Vol(cc)	Starting Gas GMW	Samp Vol(cc)	Starting Gas GMW
1.449	4	1.534	4
	1.262	1.271	

Velocity correction is for this Bucket diameter only!
Bucket Dia.(mm)
19

Offset(roughly starting wt. mg)
310.2
htga Mass (grams)
-0.06148
Head Effect Params.

m(@RTP)	b
-0.19	0

Theoretical m (htga M)-> -0.165832225 at RTP



WITNESSED SIGNATURE REDACTED

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